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Rock Island Arsenal Laboratory



TECHNICAL REPORT

COMPRESSION SET OF ELASTOMERS
AT ELEVATED TEMPERATURES

By

MAY 1 1963

E. W. Bergstrom

Dept. of the Army Project No. 1-H-0-24401-A-111

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Rock Island Arsenal
Rock Island, Illinois

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ABSTRACT

Compression set tests (constant deflection method of ASTM D395-55) were carried out on heat resistant rubber vulcanizates for periods as long as twenty-eight days at temperatures up to 500°F.

It was found that some silicone rubbers did not have set values at elevated temperatures as low as might be expected from their excellent resistance to heat aging.

Cure time was found to have a significant effect on the set of isobutylene/isoprene vulcanizates. Increased cure time resulted in lower set even though the original cure time may have been optimum for other properties.

Certain metal oxide additives significantly lowered the set of some silicones. No one additive was found, however, which lowered the set of all four types of silicones; namely, dimethyl, methyl vinyl, fluoro and high strength. Additives which had been found previously to improve the heat resistance of silicones proved to be ineffective in lowering set.

RECOMMENDATIONS

It is recommended that a combination of additives, one which improves heat resistance and one which lowers set, be incorporated in silicones required to meet both maximum heat stability and low set requirements.

It is recommended that a study be made to develop a test method and procedure for determining the ability of an elastomeric vulcanizate which has been compressed at room temperature and then subjected to high temperature, to recover from deformation when removed from the compression set device and measured while still at high temperature.

COMPRESSION SET OF ELASTOMERS AT ELEVATED TEMPERATURES

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COMPRESSION SET OF ELASTOMERS AT ELEVATED TEMPERATURES

OBJECT

The objects of this investigation were: (1) to determine the compression set at temperatures up to 500°F of the most thermally stable elastomeric vulcanizates, and (2) to improve the compression set of these vulcanizates.

INTRODUCTION

This Arsenal has previously reported^{1,2,3} data obtained on the high temperature capabilities and limitations of many types of elastomers, both after aging and when properties were measured at high temperatures. These data dealt with the effects of high temperatures on tensile, modulus, elongation, strain, hardness and percent weight loss, all of which are important. However, many rubbers are being used as gaskets or sealants where elastic recovery is an important factor. Data on compression set would be helpful in selecting vulcanizates for such applications.

During the past year, a study has been made to determine the set of heat resistant vulcanizates after they have been subjected, under compression, to temperatures up to 500°F. Attempts were also made to improve these properties. It is the purpose of this report to present the results obtained.

PROCEDURE

All compression set tests were performed using Method B (constant deflection method) of ASTM D395-55. Standard test specimens, 1/2" thick and 1.129" in diameter, were molded from compounds whose formulations are given in Table I. In order to provide equivalent states of cure for both pads and buttons, all set specimens were press cured one and one-half times longer than the press cure times given in the table for .075" thick test pads.

In some instances, various additives were used in the base formulations in attempts to improve set. This is discussed in detail in the results section of the report.

Set tests were carried out at 212°, 250°, 300°, 350°, 400°, 450° and 500° F. for periods as long as twenty-eight days. Tests were discontinued when a set value of 90 percent was reached, since it would be impractical to use a rubber having a set of 90 percent or higher.

TABLE I

COMPOUND FORMULATIONS

| COMPOUNDING INGREDIENTS | AlE | S77C | M87C | 138 | 138D | 140 | 140C3 | 148 | 148D |
|---|-------------------------------------|-------------------------------------|-------------------------------------|-------------------------------------|-------------------------------------|-------------------------------------|-------------------------------------|-------------------------------------|-------------------------------------|
| Pale Crepe | 100 | | | | | | | | |
| 76.5/23.5 Butadiene/styrene (carbon black masterbatched) | | 150 | | | | | | | |
| Butadiene/acrylonitrile (low acrylonitrile) | | | 100 | | | | | 100 | |
| Chlorinated isobutylene/isoprene | | | | | 100 | 10 | 10 | | 100 |
| Brominated isobutylene/isoprene | | | | | | 100 | 100 | | |
| Isobutylene/isoprene (2.1 - 2.5 mole % unsaturation) | | | | | | 1 | 1 | 1 | 1 |
| Stearic acid | 1 | 2 | 1 | 1 | 1 | 1 | 1 | 1 | 1 |
| Zinc oxide | 5 | 3 | 5 | 3 | 3 | 5 | 5 | 5 | 5 |
| Phenyl beta naphthylamine | 1 | 1 | | 1 | 1 | | | | |
| Tetramethyl thiuram disulfide | 3 | | | | | | | | |
| Elementary tellurium | 0.5 | | | | | | | | |
| MT Carbon Black | 25 | | | | | | | | |
| FT Carbon Black | 25 | | | | | | | | |
| Dipentamethylene thiuram tetrasulfide | | 1 | 1.5 | | | | | | |
| 2-mercaptobenzothiazole | | 1 | | | | | | | |
| MAF Carbon Black | | | 1 | | | 60 | 60 | | |
| Polymerized trimethyl dihydroquinoline | | | 50 | | | | | | |
| Magnesium oxide | | | 1 | | | | | | |
| MAF Carbon Black | | | | 2 | | | | | |
| Light processing oil | | | | 60 | | | | 50 | |
| Phenol formaldehyde resin | | | | 5 | | | | | |
| Benzothiazyl disulfide | | | | 3 | | 12.5 | 12.5 | | |
| Zinc diethyldithiocarbamate | | | | 2 | | | | 1.5 | |
| | Press cure 30 min. 63070°F | Press cure 30 min. 63070°F | Press cure 30 min. 63070°F | Press cure 30 min. 63070°F | Press cure 30 min. 63070°F | Press cure 30 min. 63200°F | Press cure 60 min. 63200°F | Press cure 45 min. 63070°F | Press cure 45 min. 63070°F |

For trade names of compounding ingredients, see Code Sheet at end of report.

TABLE I (Cont.)

| COMPOUNDING INGREDIENTS | 283 | 298 | 298T | 256C3 | 256C3T | 281 | 281P | 275DC | 285 | 2123 |
|--|--|--|--|---|---|---|---|--|--|--|
| Vinylidene fluoride/hexafluoro-propylene | 100 | | | | | | | | | |
| Dimethyl polysiloxane with vinyl groups attached - contains 20% reinforcing filler | | 100 | 100 | | | | | | | 100 |
| Methyl vinyl silicone | | | | | 100 | 100 | | | | |
| High strength silicone | | | | 100 | | | | | | |
| Fluorinated silicone | | | | | | | | 100 | | |
| Nitrile silicone | | | | | | | | | | |
| Dimethyl silicone | 20 | | | | | | | | | |
| KT Carbon Black | 15 | | | | | | | | 100 | |
| Magnesium oxide | | | | | | | | | | |
| N,N'-Dicinnamylidene-1,6-hexanediamine | 3 | 8 | 8 | | | | 10 | | | 40 |
| Precipitated silica | | 23 | 23 | | | | | | | |
| Ground quartz | | | | | | | | | | |
| 50% Dichlorobenzoyl peroxide with silicone fluid | | 1.6 | 1.6 | 1.3 | 1.3 | 1.3 | 1.3 | | 2.4 | |
| 50% Benzoyl peroxide with silicone fluid | | | 2 | | 2 | | | 0.8 | | |
| Ferric oxide | | | | | | | | | | |
| Tertiary butyl peroxide | | | | | | | | | | |
| | Press cure 30 min. @307°F Step cure in air oven to 400°F Postcure 24 hrs. @400°F | Press cure 5 min. @240°F Postcure 8 hrs. @480°F | Press cure 5 min. @240°F Postcure 8 hrs. @480°F | Press cure 10 min. @275°F Postcure 8 hrs. @375°F | Press cure 10 min. @275°F Postcure 8 hrs. @375°F | Press cure 5 min. @240°F Postcure 24 hrs. @300°F | Press cure 5 min. @240°F Postcure 24 hrs. @300°F | Press cure 20 min. @340°F Postcure 24 hrs. @350°F | Press cure 10 min. @240°F Postcure 24 hrs. @480°F | Press cure 20 min. @340°F Postcure 24 hrs. @480°F |

RESULTS

The first phase of this investigation dealt with the determination of set on vulcanizates which had been found previously by this Arsenal to possess good heat resistance, namely, various silicones and isobutylene/isoprene vulcanizates (including the brominated and chlorinated copolymers) and a vinylidene fluoride/hexafluoropropylene vulcanizate. NR, SBR and NBR elastomers were compounded for good aging resistance and used as control vulcanizates. Duplicate test results agreed within one or two percent in most cases and never varied by more than four percent. The results given in Table II reveal the following:

1. In some cases there is little relationship between the heat resistance of a vulcanizate (as measured by retention of tensile, modulus, elongation and strain) and its set. For example, vulcanizates which employ a phenol formaldehyde resin - tetramethyl thiuram disulfide - benzothiazyl disulfide curing system (I38 and I38D) have excellent heat resistance at elevated temperatures in comparison with other butyl vulcanizates², but have very high set at temperatures of only 212° and 250°F. On the other hand, vulcanizates prepared from compounds employing a zinc diethyldithiocarbamate curing system (I48 and I48D) have heat resistance almost identical to those employing the resin cure, but have set values that are much superior (lower). The set of the chlorinated vulcanizate (I48) is very good, being lower than some of the silicones.

2. Although cure time does not make a significant difference in the heat resistance of isobutylene/isoprene elastomers (based on percent change in tensile, modulus, elongation and strain) it may have a very pronounced effect on set. Vulcanizates of compound I40C3 were cured twice as long as those of compound I40. The heat resistance of these compounds are very similar as shown in Table III. The set at temperatures of 300°F and above, however, is much better for I40C3 than for I40. The cure time for I40 is considered to be optimum⁴. It appears from these data, however, that the cure time considered to be optimum for other properties may not give the lowest compression set.

3. The silicones did not have as good recovery at the higher temperatures as might be expected from their excellent thermal stability. This is especially true of the high strength silicone which had reached 100% set after 8 hours at 450°F.

COMPRESSION SET (PERCENT) OF VARIOUS VULCANIZATES AT HIGH TEMPERATURES

5

TABLE II (Cont.)

| RIA CPD. NO. | ELASTOMER | 22 IR. 350F | 70 HR. 350F | 7 DAYS 350F | 14 DAYS 350F | 28 DAYS 350F | 8 HR. 400F | 16 HR. 400F | 22 HR. 400F | 70 HR. 400F | 7 DAYS 400F | 14 DAYS 400F | 8 HR. 450F | 16 HR. 450F | 22 HR. 450F | 70 HR. 450F | 8 HR. 500F | 16 HR. 500F |
|--------------------|--|-------------------|-------------------|-------------------|--------------------|--------------------|------------------|-------------------|-------------------|-------------------|-------------------|--------------------|------------------|-------------------|-------------------|-------------------|------------------|-------------------|
| A1E | Pale Crope | 75 | 97 | - | - | - | 92 | - | - | - | - | - | - | - | - | - | - | - |
| S77C | Butadiene/styrene | 80 | 112 | - | - | - | 94 | - | - | - | - | - | - | - | - | - | - | - |
| N87C | Butadiene/acrylo- nitrile | 72 | 99 | - | - | - | 83 | 94 | - | - | - | - | - | - | - | - | - | - |
| I40 | Isobutylene/isoprene | 87 | 94 | - | - | - | 89 | 93 | - | - | - | - | - | - | - | - | - | - |
| I40C3 | Isobutylene/isoprene | 73 | 77 | 81 | 86 | 89 | 72 | 76 | 76 | 80 | 85 | 92 | 82 | 83 | 86 | 92 | - | - |
| I48 | Chlorinated Isobuty- lene/isoprene | 52 | 71 | 82 | 88 | 98 | 64 | 73 | 79 | 89 | 95 | - | 88 | 100 | - | - | - | - |
| I48D | Brominated Isobuty- lene/isoprene | 76 | 99 | - | - | - | 84 | 96 | - | - | - | - | 111 | - | - | - | - | - |
| Z98 | Methyl vinyl silicone | 49 | 68 | 86 | 92 | - | 34 | 62 | 70 | 93 | - | - | 88 | 106 | - | - | 122 | - |
| Z98T | Methyl vinyl silicone | 43 | 73 | 86 | 92 | - | 33 | 63 | 67 | 95 | - | - | 81 | 103 | - | - | 119 | - |
| Z56C3 | High strength silicone | 70 | 90 | - | - | - | 83 | 98 | - | - | - | - | 105 | - | - | - | - | - |
| Z56C3T | High strength silicone | 72 | 91 | - | - | - | 86 | 98 | - | - | - | - | 100 | - | - | - | - | - |
| Z81 | Fluoro silicone | 24 | 51 | 67 | 79 | 95 | 31 | 45 | 54 | 90 | - | - | 57 | 86 | 100 | - | 109 | - |
| Z83 | Vinylidene fluoride/ hexafluorocyclopentene | 38 | 55 | 61 | 73 | 75 | 33 | 46 | 55 | 72 | 83 | 92 | 52 | 73 | 75 | 94 | 84 | 100 |

TABLE III

**PERCENT CHANGE IN PHYSICAL PROPERTIES OF ISOBUTYLENE/ISOPRENE
VULCANIZATES AFTER AIR OVEN EXPOSURE**

| PHYSICAL PROPERTIES | ORIGINAL | 8 HR. | 8 HR. | 8 HR. | 8 HR. | 8 HR. | 8 HR. | 8 HR. |
|--|----------|-------|-------|-------|-------|-------|-------|----------------|
| | | ②212F | ②250F | ③300F | ③350F | ④400F | ④450F | ⑤500F |
| Tensile | 1690 psi | +4 | +4 | +5 | -11 | -20 | -41 | -88 |
| Elongation | 580% | -5 | -19 | -26 | -38 | -43 | -36 | +17 |
| Strain (400 psi load for 60 sec) | 174 | -8 | -20 | -26 | -26 | -24 | +7 | Broke |
| I40 | | | | | | | | |
| Tensile | 1710 psi | +9 | -2 | -3 | -12 | -18 | -47 | Speci- mens |
| Elongation | 510% | -14 | -22 | -33 | -35 | -43 | -41 | had no |
| Strain (400 psi load for 60 sec) | 133 | -8 | -13 | -19 | -22 | -20 | +8 | strength |
| I40C3 | | | | | | | | |

4. The addition of ferric oxide to methyl vinyl and high strength silicones has little or no effect on the set. It has long been recognized^{5,6,7,8} that the addition of ferric oxide improved the thermal stability.

5. A vinylidene fluoride/hexafluoropropylene vulcanizate (Z83) was the only one which had a value below 90% after 8 hours at 500°F. This rubber also had the lowest set when exposed 14 days at 300°F, 7 days at 350°F, 70 hours at 400°F or 8 hours at 450°F.

Previous work³ revealed that several additives had improved the thermal stability of a methyl vinyl silicone. The results presented in Table II, however, show that ferric oxide, one of the most effective additives for improving the heat resistance of a methyl vinyl silicone (Z98), does not improve the set of this vulcanizate. It was revealed in the literature that mercuric oxide⁵ and cadmium oxide^{6,10} improve the set of silicones. However, it was found by this Arsenal³ that neither of these chemicals improved the resistance to high temperatures of a methyl vinyl silicone. It was therefore decided to re-evaluate all those additives which did not degrade the original physical properties of the Z98 silicone. Some additional additives were also used, a complete list being furnished in the Appendix. These additives were evaluated at a concentration of two parts unless otherwise indicated. Seven (barium oxide, cadmium oxide, calcium oxide, calcium hydroxide, magnesium oxide, praseodymium oxide and strontium oxide) were found to lower the set of a methyl vinyl silicone. These data are given in Table IV.

It was found that these additives were not as effective, however, in this vulcanizate (Z98) when the buttons were postcured 24 hours at 480°F instead of 8 hours at 480°F. The 24 hour postcure reduces the set of the control as shown below:

| | Exposed 7 days @300°F | Exposed 70 hr. @350°F | Exposed 22 hr. @400°F | Exposed 8 hr. @450°F |
|-------------------------|-----------------------------|-----------------------------|-----------------------------|----------------------------|
| Postcured 8 hr. @480°F | 63 | 68 | 70 | 88 |
| Postcured 24 hr. @480°F | 35 | 48 | 45 | 46 |

Cadmium oxide was the only additive which significantly lowered the set after a postcure of 24 hours at 480°F. This is shown in Table V where samples based on the two postcures have been exposed at 300°F. The manufacturer of this silicone

TABLE IV
IMPROVEMENT IN SET OF A METHYL VINYL SILICONE
BY VARIOUS ADDITIVES

| <u>Additive</u> | <u>F.rts/ 100 rhc</u> | <u>7 Days 212F</u> | <u>7 Days 300F</u> | <u>70 Hour 350F</u> | <u>22 Hour 400F</u> | <u>8 Hour 450F</u> | <u>8 Hour 500F</u> |
|---------------------|-------------------------------|----------------------------|----------------------------|-----------------------------|-----------------------------|----------------------------|----------------------------|
| None - Control | - | 39 | 63 | 68 | 70 | 88 | 122 |
| Barium oxide | 2 | 30 | 52 | 45 | 39 | 37 | 73 |
| Cadmium oxide | 2 | 19 | 40 | 50 | 41 | 41 | 113 |
| Calcium hydroxide | 2 | 21 | 43 | 44 | 34 | 40 | 88 |
| Calcium oxide | 2 | 24 | 44 | 48 | 45 | 41 | 94 |
| Magnesium oxide | 2 | 21 | 40 | 35 | 34 | 32 | 73 |
| Praeseodymium oxide | 2 | 29 | 41 | 38 | 41 | 39 | 84 |
| Strontium oxide | 2 | 19 | 51 | 44 | 39 | 35 | 75 |

TABLE V
EFFECT OF POSTCURE TIME ON EFFECTIVENESS OF ADDITIVES
FOR LOWERING SET IN METHYL VINYL SILICONE

| <u>Additive</u> | <u>Exposed 22 hrs. @300F</u> | | <u>Exposed 70 hrs. @300F</u> | |
|---------------------|---|--|---|--|
| | <u>Postcured 8 hours @480°F</u> | <u>Postcured 24 hours @480°F</u> | <u>Postcured 8 hours @480°F</u> | <u>Postcured 24 hours @480°F</u> |
| None - Control | 33 | 16 | 50 | 31 |
| Barium oxide | 17 | 14 | 31 | 30 |
| Calcium hydroxide | 17 | 14 | 31 | 29 |
| Calcium oxide | 19 | 20 | 29 | 30 |
| Cadmium oxide | 8 | 6 | 22 | 10 |
| Magnesium oxide | 22 | 14 | 40 | 26 |
| Praeseodymium oxide | 23 | 15 | 38 | 31 |
| Strontium oxide | 19 | 15 | 31 | 28 |

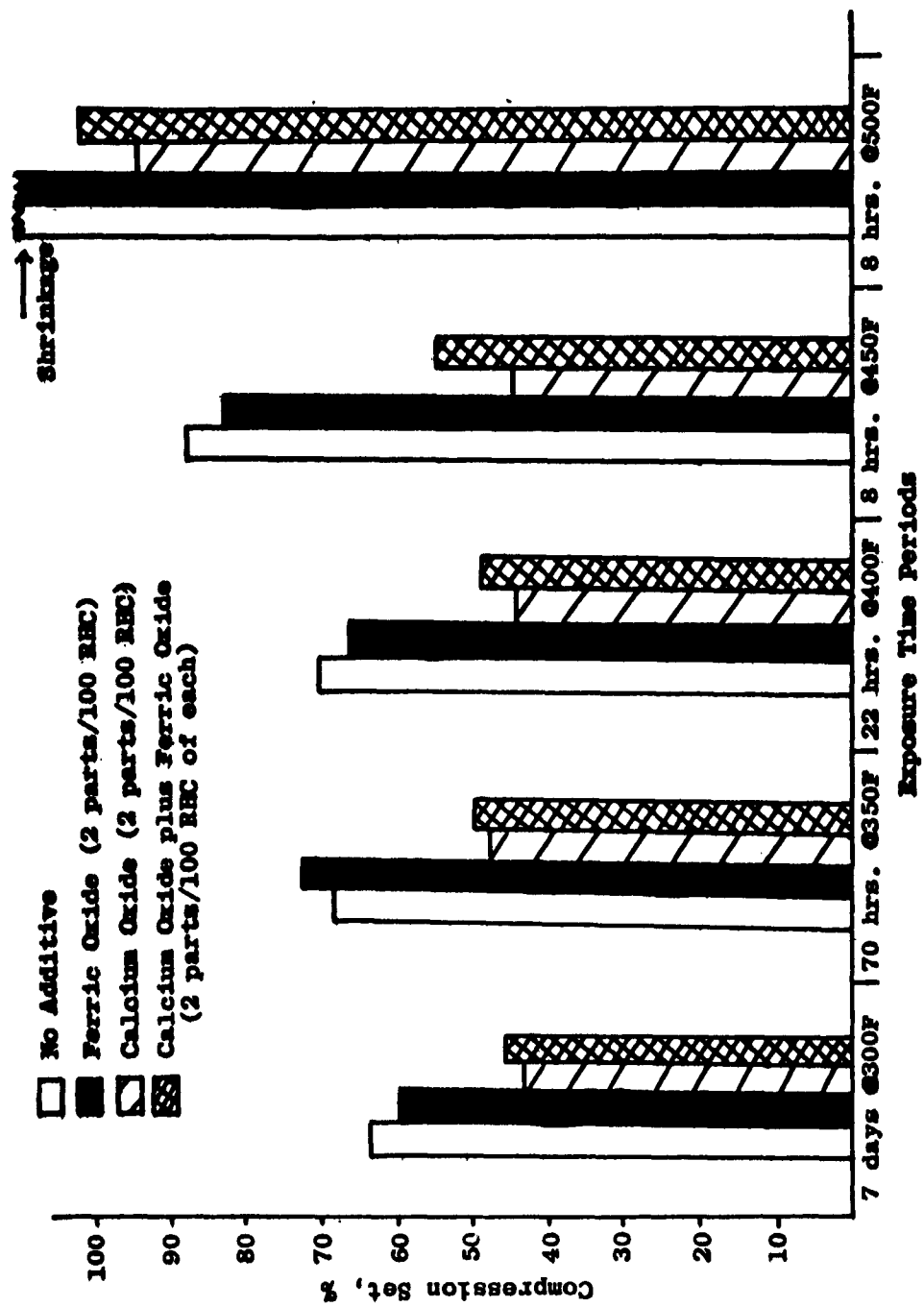
recommends⁸ oven postcures of 4 to 24 hours at 480°F to develop an optimum balance of properties. The postcure time is varied from 4 to 24 hours to obtain different combinations of properties to meet specific specification or job requirements. The postcure used most frequently in testing the Z98 vulcanizate was 8 hours at 480°F since this was the time recommended⁹. The 8 hour postcure gives a 50±5 Shore A hardness while the 24 hour postcure gives 60±5. It can be seen in Table V that the set at 300°F of the controls postcured 24 hours at 480°F are lower than those postcured 8 hours at 480°F. The use of additives in the vulcanizates postcured 8 hours at 480°F lowered the set in many cases to the same range as that of those postcured 24 hours at 480°F. Also, the good high temperature resistance was not adversely affected at temperatures of 600°F and 700°F by the longer postcure time (24 hours vs. 8 hours). The Z98T compound is the most heat resistant silicone found by this Arsenal.

None of the additives which improved the thermal stability of the Z98 were effective in improving the set. Likewise, none of the seven additives which lowered the set of vulcanizates postcured 8 hours at 480°F improved the heat resistance. Concentrations of 5 and 10 parts of the additives did not improve the set over those containing only two parts. However, those containing two parts had lower set in most cases than those containing one.

Various combinations of additives found to be effective in improving heat resistance and in lowering set were evaluated in a methyl vinyl silicone. It was found that a vulcanizate containing a combination of two parts ferric oxide and two parts calcium oxide had both improved heat resistance and improved set. The improvement in set is shown graphically in Figure 1, and the improvement in heat resistance is shown in Table VI.

A study was also made to determine whether those additives found to be effective in lowering the set of a methyl vinyl silicone postcured 8 hours at 480°F (Z98) would be as effective in improving the set of a high strength (Z56C3), fluoro (Z81F), and dimethyl (Z85) silicone and another methyl vinyl silicone compounded for low set (Z123). The results of this evaluation are given in Table VII and reveal the following:

1. Some of the additives were not as effective in lowering the set of the high strength, fluoro and dimethyl silicones as they were in lowering that of the methyl vinyl silicone. Cadmium oxide, for example, was the only additive which significantly improved the set of the dimethyl silicone.



COMPRESSION SET OF SILICONE VULCANIZATES CONTAINING
CALCIUM OXIDE AND/OR FERRIC OXIDE

TABLE VI
HEAT RESISTANCE OF SILICONE VULCANIZATES CONTAINING
CALCIUM OXIDE AND/OR FERRIC OXIDE

| PHYSICAL PROPERTIES | NO ADDITIVE - CONTROL | | | | FERRIC OXIDE (2 PARTS) | | | |
|------------------------|-----------------------|-------------------|------------------|------------------|------------------------|-------------------|------------------|------------------|
| | ORIGINAL | AGED | | TESTED @300°F | PHYSICAL PROPERTIES | AGED | | |
| | | 70 HRS. @600°F | 7 DAYS @600°F | | | 70 HRS. @600°F | 7 DAYS @600°F | 2 HRS. @700°F |
| T | 840 | 500 | Brittle | 580 | T | 780 | 400 | 220 |
| 100% M | 120 | - | " | 200 | 100% M | 130 | - | - |
| 200% M | 410 | - | " | 490 | 200% M | 450 | - | - |
| 300% M | 680 | - | " | - | 300% M | 680 | - | - |
| % E | 420 | 50 | " | 230 | % E | 360 | 80 | 50 |
| H | 50 | 86 | " | - | H | 51 | 75 | 71 |
| | | | | | | | | 570 |
| | | | | | | | | 220 |
| | | | | | | | | 230 |
| | | | | | | | | 480 |
| | | | | | | | | - |
| | | | | | | | | 230 |
| | | | | | | | | - |

| PHYSICAL PROPERTIES | CALCIUM OXIDE (2 PARTS) | | | | CALCIUM OXIDE PLUS FERRIC OXIDE (2 PARTS) | | | |
|------------------------|-------------------------|-------------------|------------------|------------------|---|-------------------|------------------|------------------|
| | ORIGINAL | AGED | | TESTED @300°F | PHYSICAL PROPERTIES | AGED | | |
| | | 70 HRS. @600°F | 7 DAYS @600°F | | | 70 HRS. @600°F | 7 DAYS @600°F | 2 HRS. @700°F |
| T | 610 | 520 | Brittle | 460 | T | 840 | 600 | 350 |
| 100% M | 130 | - | " | 170 | 100% M | 200 | - | - |
| 200% M | 360 | - | " | 370 | 200% M | 540 | - | - |
| 300% M | 560 | - | " | - | 300% M | 710 | - | - |
| % E | 350 | 50 | " | 230 | % E | 320 | 100 | 70 |
| H | 48 | 85 | " | - | H | 55 | 77 | 85 |
| | | | | | | | | 600 |
| | | | | | | | | 260 |
| | | | | | | | | 580 |
| | | | | | | | | - |
| | | | | | | | | 210 |
| | | | | | | | | - |
| | | | | | | | | 78 |

Note: T = Tensile, psi
M = Modulus, psi
E = Ultimate Elongation, %
H = Hardness, Shore A

TABLE VII

EFFECTIVENESS OF ADDITIVES IN IMPROVING
SET OF VARIOUS SILICONESHigh Strength Silicone (Z56C3)

| Additive | Parts/ 100 rhc | 70 Hrs @250°F | 70 Hrs @300°F | 22 Hrs @350°F | 8 Hrs @400°F |
|---------------------|-------------------|------------------|------------------|------------------|-----------------|
| None - Control | - | 38 | 63 | 70 | 83 |
| Barium oxide | 2 | 39 | 52 | 50 | 52 |
| Cadmium oxide | 2 | 31 | 54 | 58 | 63 |
| Calcium hydroxide | 2 | 28 | 43 | 50 | 55 |
| Calcium oxide | 2 | 33 | 48 | 59 | 59 |
| Magnesium oxide | 2 | 32 | 57 | 54 | 56 |
| Praeseodymium oxide | 2 | 35 | 49 | 52 | 49 |
| Strontium oxide | 2 | 21 | 42 | 47 | 50 |

Fluoro Silicone (Z81F)

| Additive | Parts/ 100 rhc | 7 Days @300°F | 70 Hrs @350°F | 22 Hrs @400°F | 16 Hrs @450°F |
|---------------------|-------------------|------------------|------------------|------------------|------------------|
| None - Control | - | 26 | 25 | 39 | 92 |
| Barium oxide | 2 | 13 | 14 | 18 | 47 |
| Cadmium oxide | 2 | 21 | 22 | 38 | 79 |
| Calcium hydroxide | 2 | 15 | 10 | 17 | 53 |
| Calcium oxide | 2 | 17 | 19 | 23 | 55 |
| Magnesium oxide | 2 | 26 | 21 | 26 | 41 |
| Praeseodymium oxide | 2 | 26 | 24 | 21 | 51 |
| Strontium oxide | 2 | 14 | 10 | 12 | 68 |

Dimethyl Silicone (Z85)

| Additive | Parts/ 100 rhc | 70 Hrs @212°F | 70 Hrs @250°F | 70 Hrs @300°F | 8 Hrs @350°F |
|---------------------|-------------------|------------------|------------------|------------------|-----------------|
| None - Control | - | 69 | 78 | 85 | 72 |
| Barium oxide | 2 | | Not Compatible | | |
| Cadmium oxide | 2 | 19 | 27 | 48 | 18 |
| Calcium hydroxide | 2 | 69 | 76 | 86 | 64 |
| Calcium oxide | 2 | 62 | 65 | 74 | 57 |
| Magnesium oxide | 2 | | Not Compatible | | |
| Praeseodymium oxide | 2 | 66 | 76 | 79 | 68 |
| Strontium oxide | 2 | 67 | 76 | 84 | 72 |

TABLE VII (Cont.)

Methyl Vinyl Silicone Vulcanizate (Z123)

| <u>Additive</u> | <u>7 Days @250°F</u> | <u>70 Hrs @300°F</u> | <u>22 Hrs @350°F</u> | <u>22 Hrs @400°F</u> | <u>8 Hrs @500°F</u> |
|-------------------|--------------------------|--------------------------|--------------------------|--------------------------|-------------------------|
| None - Control | 20 | 17 | 16 | 22 | 43 |
| Barium oxide | 17 | 19 | 17 | 21 | 35 |
| Calcium hydroxide | 17 | 17 | 15 | 18 | 37 |
| Calcium oxide | 17 | 15 | 16 | 19 | 34 |
| Cadmium oxide | 17 | 14 | 13 | 21 | 39 |
| Magnesium oxide | 18 | 17 | 15 | 19 | 38 |
| Strontium oxide | 19 | 16 | 15 | 18 | 35 |

2. There is no one additive which improves the set of all four types of silicones studied. However, the set of compounds Z56C3, Z81F, and Z85 could be improved by one or more additives.

3. Compound Z123 is a methyl vinyl silicone especially compounded for low set, and it is to be noticed that its set is very low at high temperatures. None of the additives significantly improved the set of this vulcanizate. However, even when ferric oxide is added to this compound it does not have as good heat resistance as the other methyl vinyl silicone studied, Z98T. Specimens of Z123 containing 2 parts ferric oxide are brittle after exposure for 7 days at 600°F or two hours at 700°F in an air oven whereas Z98T specimens are still flexible after similar exposure.

Calcium oxide and cadmium oxide were evaluated in SBR (S77C), NBR (N87C), NR (A1E), IIR (I40C3), chlorobutyl (I48), bromobutyl (I48D) and vinylidene fluoride/hexafluoropropylene (Z83) vulcanizates to determine if the set of these vulcanizates could be lowered. The only vulcanizate which showed significant improvement in set without having its original physical properties affected was the NBR (N87C) containing two parts calcium oxide. The improvement in set is shown in Table VIII. The addition of two parts cadmium oxide to the SBR and NBR vulcanizates produced lower set (Table X), but the original physical properties were altered as shown in Table IX.

TABLE VIII

IMPROVEMENT IN THE SET OF NBR BY CALCIUM OXIDE

| <u>Additive</u> | <u>Parts/ 100 rhc</u> | <u>70 Hrs @212°F</u> | <u>22 Hrs @250°F</u> | <u>22 Hrs @300°F</u> | <u>22 Hrs @350°F</u> |
|-----------------|-------------------------------|--------------------------|--------------------------|--------------------------|--------------------------|
| None - Control | - | 17 | 23 | 38 | 72 |
| Calcium oxide | 2 | 15 | 14 | 27 | 56 |

TABLE IX

PHYSICAL PROPERTIES OF SBR AND NBR CONTAINING CADMIUM OXIDE

| <u>Physical Property</u> | <u>SBR</u> | | <u>NBR</u> | |
|------------------------------|----------------------------------|--------------------------|----------------------------------|--------------------------|
| | <u>No Additive (Control)</u> | <u>Cadmium Oxide</u> | <u>No Additive (Control)</u> | <u>Cadmium Oxide</u> |
| Tensile, psi | 3150 | 2680 | 2510 | 2610 |
| 100% Modulus, psi | 240 | 430 | 300 | 740 |
| 200% Modulus, psi | 510 | 1100 | 880 | 2110 |
| 300% Modulus, psi | 1040 | 2070 | 1790 | - |
| Ultimate Elong- ation, % | 680 | 370 | 400 | 250 |
| Hardness, Shore A | 65 | 66 | 63 | 70 |

TABLE X

SET OF SBR AND NBR CONTAINING CADMIUM OXIDE

| | | Parts/ 100 rhc | 70 Hrs @212°F | 22 Hrs @250°F | 22 Hrs @300°F | 22 Hrs @350°F |
|----------|----------------|----------------------|------------------|------------------|------------------|------------------|
| Additive | | | | | | |
| SBR | None - Control | - | 36 | 46 | 61 | 80 |
| | Cadmium oxide | 2 | 22 | 20 | 31 | 54 |
| NBR | None - Control | - | 17 | 23 | 38 | 72 |
| | Cadmium oxide | 2 | 7 | 6 | 17 | 37 |

The NR compound containing cadmium oxide did not cure. The addition of calcium oxide to the NR had no effect on either the physical properties or the set.

Both the calcium and cadmium oxides retarded the cure of the butyl vulcanizates (I48, I48D, and I40C3). This retardation of cure was very pronounced when the calcium oxide was used. The set of the vulcanizates containing the calcium and cadmium oxides were generally poorer than the control compound containing no additive.

The addition of the calcium and cadmium oxides to the vinylidene fluoride/hexafluoropropylene vulcanizates had no effect on either the original physical properties or set.

DISCUSSION

It was found that calcium, cadmium, praseodymium, barium, magnesium and strontium oxides improved the set of some silicone vulcanizates at elevated temperatures. It is interesting to note that all of the metals of the effective oxides, except praseodymium, fall in group II of the periodic listing of the elements. Mercury, the oxide of which is often quoted in the literature as improving the set of silicones, also falls in the group II classification (this additive when evaluated in the Z98 formulation, however, caused the specimens to become porous and puffy during postcure, and set could not be determine).

Many of the high temperature rubbers available today are used as compression gaskets at elevated temperatures. If the gaskets are cooled to ambient temperatures before being relieved from the compression deformation, their ability to recover from the deformation can be measured by the ASTM D395 procedure. If, however, the compression is released with the gasket at the high exposure temperature, the ASTM procedure becomes inadequate. It is the opinion of this Arsenal that a test method should be developed for determining the set of vulcanizates which are: (1) tested at high temperature, (2) allowed to recover from deformation at high temperature, and (3) measured at high temperature. This should be an additional compression set test and not a replacement for the currently used ASTM method since it is felt that a knowledge of the compression set of vulcanizates which have been tested at high temperatures and then allowed to recover at room temperature is also very useful for many applications.

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APPENDIX

Additives Evaluated to Improve the Compression Set Properties of a Methyl Vinyl Silicone Vulcanizate

| | |
|-----------------------|--------------------------------------|
| Antimony trioxide | Lanthanum oxide |
| Barium oxide | Magnesium oxide |
| Barium zirconate | Manganese dioxide* (1) |
| Beryllium oxide | Mercury oxide* |
| Cadmium oxide | Molybdenum oxide |
| Calcium carbonate | Neodymium oxide |
| Calcium hydroxide | Nickel oxide |
| Calcium oxide | P-33 Carbon black* (1) |
| Cerium oxide | Platinum oxide* |
| Chromium oxide | Praeseodymium oxide |
| Cobalt oxide | Scandium oxide |
| Copper oxide | Silver oxide |
| Erbium oxide | Stannic oxide |
| Europium oxide | Strontium oxide |
| Ferric fluoride* | Tantalum pentoxide |
| Ferric formate* | Terbium oxide |
| Ferric octoate* (1) | Thorium oxide |
| Ferric oxide | Thulium oxide |
| Ferric pyrophosphate* | Titanium oxide |
| Ferric sulfide | Ytterbium oxide |
| Gadolinium oxide | Zinc oxide |
| Gallium oxide | Zirconium oxide |
| Hafnium oxide | Zirconium silicate |
| Holmium oxide | 2,5 ditertiary butyl-p-benzoquinone* |
| Iron phosphate (ins.) | |
| Iron silicate* | |

*Compression set buttons become porous and puffy during postcure - set values were, therefore, not determined.

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| Brominated Isobutylene/ isoprene | Hycar 2202 | B.F. Goodrich Chem. Company |
| Isobutylene/isoprene (2.1-2.5 mole % unsat- uration) | 325 Butyl | Enjay Company, Inc. |
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| 2-mercaptobenzothiazole | Captax | R.T. Vanderbilt Co. |
| MAF Carbon black | Philblack A | Phillips Petroleum Co. |
| Polymerized trimethyl dihydroquinoline | Agerite Resin D | R.T. Vanderbilt Co. |
| HAF Carbon black | Philblack O | Phillips Petroleum Co. |
| Light Processing Oil | Circo Light Processing Oil | Sun Oil Company |
| Phenol formaldehyde resin | Amberol ST-137 | Rohm & Haas Co. |
| Benzothiazyl disulfide | Altax | R.T. Vanderbilt Co. |
| Zinc diethyldithio- carbamate | Ethyl Zimate | R.T. Vanderbilt Co. |
| N,N'-Dicinnamylidene- 1,6-hexanediamine | Diak #3 | E.I. duPont de Nemours & Co. |
| Precipitated silica | Hi Sil X303 | Columbia-Southern Chemical Corp. |
| Ground quartz | Neo Novacite | Malvern Minerals Co. |
| 50% Dichlorobenzoyl peroxide with silicone fluid | Cadox T.S. Paste | Cadet Chemical Corp. |
| 50% Benzoyl peroxide with silicone fluid | Cadox S.G. Paste | Cadet Chemical Corp. |
| Dimethyl silicone | SE-30 | General Electric |

| | | |
|--|---|---|
| <p>AD Rock Island Arsenal Laboratory, Rock Island, Illinois COMPRESSION SET OF ELASTOMERS AT ELEVATED TEMPERATURES, by E. W. Bergstrom</p> <p>RIA Lab. Rep. 63-798, 14 Mar 63, 24 p. incl. illus. tables, (DA Project No. 1-H-0-24401- A-111, AMC Code No. 5026.11.843) Unclassified report.</p> <p>Compression set tests (constant deflection method of ASTM D395-55) were carried out on heat resistant rubber vulcanizates for periods as long as 28 days at temperatures up to 500°F. It was found that some sil- cone rubbers did not have set values at elevated temperatures as low as might be expected from their excellent resistance to heat aging. Cure time was found to have a significant effect on the set of isobutylene/ isoprene vulcanizates. Increased cure time resulted in lower set even though the origi- nal cure time may have been optimum for other properties.</p> <p>(Cont.) over</p> | <p>UNCLASSIFIED</p> <p>1. Rubber-Heat Stability</p> <p>2. Rubber-Test Results</p> <p>DISTRIBUTION: Copies obtainable from ASTIA</p> | <p>UNCLASSIFIED</p> <p>1. Rubber-Heat Stability</p> <p>2. Rubber-Test Results</p> <p>DISTRIBUTION: Copies obtainable from ASTIA</p> |
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